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(54) Title: POLISHING COMPOSITION AND METHOD

(57) Abstract: To provide a polishing composition which enables maintenance of excellent properties and high quality of the surface of a hard disk without lowering polishing rate during polishing of the surface, and which can provide a polished surface in which the amount of dub-off is considerably reduced as compared with that of a conventional level, a polishing composition containing water, a polishing material (particularly alumina), a polishing accelerator, and at least one of hydroxypropyl cellulose and hydroxyalkyl alkyl cellulose is provided.

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5 TECHNICAL FIELD

5 The present invention relates to a polishing composition which is employed for precise polishing of metal, plastic, or glass, particularly for fine polishing of high-quality mirror-surface polishing.

10 The present invention is employed for precise polishing of the surface of an aluminum magnetic disk (hereinafter the disk will be referred to as a "hard disk") which is installed in a hard disk drive of a computer.

15 In recent years, as high-performance computers have been developed and computers have been downsized, there has been demand for high-quality mirror-surface polishing which in hard disks without surface defects, in accordance with an increase in recording density of the disks. In order to meet such demand for surface polishing, there have been attained a variety of technical developments on

20 for example, Japanese Patent Application Laid-Open (kokai) No. 62-25187 discloses a polishing composition containing an inorganic salt, serving as a polishing accelerator, such as nickel nitrate or aluminum nitrate for increasing polishing rate. Japanese Patent

25 for increasing polishing rate, such as a lactic acid such as gluconic acid or lactic acid, and a sodium salt such as glycocantyl lactate, and a sodium salt

30 therefore for increasing polishing rate, and which can provide a polished surface with reduced surface defects.

35 Japanese Patent Application Laid-Open (kokai) No. 7-216345 discloses a polishing composition which contains an organic acid, a molybdate acid salt, and alumina so as to attain a high polishing rate and a polished surface with reduced surface defects. These polishing compositions have been developed in order to maintain

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high rate of polishing a hard disk, reduce surface roughness and surface defects, and increase recording density.

Meanwhile, Japanese Patent Application Laid-Open (kokai) Nos. 5-2747 and 5-89459 disclose methods for reducing dub-off at the circumferential end of a hard disk and for increasing recording area, in order to increase recording capacity per hard disk. However, these publications do not disclose a polishing composition, although they disclose conditions for polishing. Japanese Patent Application Laid-Open (kokai) No. 1-263186 discloses a polishing composition containing triethanolamine carboxylic acid, triethanol hydrochloride, and aluminum stearate for reducing the amount of dub-off. However, since hard disks these days must meet very strict requirements concerning surface roughness, such a polishing composition cannot be directly applied to high-precision finishing of polished surfaces.

The polishing compositions disclosed in the above publications have been developed in order to enhance polishing rate, to reduce surface defects such as micro-pits and micro-protrusions as well as scratches on the surface of a hard disk for improvement of quality, and to reduce surface roughness for increasing recording density. Incidentally, there has been demand for increasing recording capacity in a hard disk of conventional size. In order to increase recording capacity of a hard disk, recording density per unit area in the disk must be increased. However, during polishing of a hard disk, the circumference of the disk is excessively polished to form a curve portion. Such an unavoidable curve portion is called "dub-off" or "roll-off," and a region containing dub-off in a hard disk cannot be employed for recording. If the amount of dub-off can be reduced as much as possible, recording capacity per hard disk can be increased. Therefore,

B: Point on the curve which is 2,000 μm from perpendicular line h

C: Point on a linear line passing through points A and B, which is 500 μm from perpendicular line h

5 k: Perpendicular line passing through point C

 D: Point at which perpendicular line k and curve S cross

 t: Length between point C and point D (the amount of dub-off)

10 Best Modes for Carrying Out the Invention

An unavoidable phenomenon during polishing by use of free abrasive grains is occurrence of dub-off in a polished disk. The mechanism of dub-off is not necessarily clarified. However, through performing 15 polishing operation over years, it has been found that when polishing rate is high, the amount of dub-off of a disk is reduced, but surface roughness generally increases and protrusions tend to be generated on the disk; and that when polishing rate is low, the amount of 20 dub-off of a disk increases and pits tend to be generated on the disk. Meanwhile, it has been found that when a disk sinks deeply into a polishing pad, the amount of dub-off of the disk tends to increase. On the basis of 25 these findings, extensive studies have been performed on a variety of additives, for example, in order to increase the viscosity of a polishing solution while the performance of a polishing material contained in the solution is maintained. The polishing composition of the present invention has been accomplished on the basis of 30 the studies.

In the present invention, the amount of dub-off is determined as follows, as described with reference to Fig. 1.

35 As shown in Fig. 1, a circumferential portion of a polished hard disk is traced along the surface by use of a surfcoorder to draw a curve S. A perpendicular line h

is drawn along the circumferential end of the curve S. Points on the curve S which are 3,000 nm and 2,000 nm from the perpendicularly line h towards the center of the disk are assigned A and B, respectively. On a linear line passing the points A and B, a point which is 500 nm from the perpendicularly line h is assigned C. A point C and D is determined as the amount of dub-off of curve S across is assigned D. The length between the and a point at which the perpendicular line k and the perpendicularly line k is drawn so as to pass the point C, consequently, it was found that when hydroxyl methyl cellulose (HMC) or hydroxylethyl methyl cellulose (HEMC), or ethyl hydroxyethyl cellulose hydroxyl methyl cellulose (HMC), or hydroxylethyl cellulose (HEC), which has a more sterical structure among water-soluble cellulose derivatives, is added to a polymer an excellent polished surface with a small amount of dub-off while high polishing rate and high surface accuracy are maintained.

25 The mechanism of reduction in the amount of dub-off through the addition of cellulose may be attributed to the end group of cellulose ether and thickening type of cellulose ether and thickening yet been elucidated, but the molecular structure or the through the addition of HPC, HPMC, or EHEC has not been elucidated.

30 No particular limitation is imposed on the form of crystall structure, such as, α , β , or γ , of alumina which is preferably employed as a polishing material in the present invention, but α -alumina is more preferable in alumina is determined according to the desired surface consideration of polishing rate. The particle size of alumina is determined according to the desired surface 35

roughness of a disk. The mean particle size of alumina is generally 0.02-5 μm , preferably 0.1-3 μm . The particle size distribution of alumina may be preferably as narrow as possible. The amount of alumina may be 1-30
5 wt.% on the basis of the entirety of a polishing composition, preferably 3-20 wt.%.

A polishing material which is employed in the present invention is not limited to alumina, and silica, titania, zirconia, or cerium oxide may be employed to
10 obtain an effect similar to that of alumina. These polishing materials may be employed in combination.

The particle size and the amount of the polishing material which is employed may be determined in a manner similar to the case in which alumina is employed, but
15 particle size and amount may be changed.

A polishing accelerator which may be employed in the present invention may be an organic acid or an inorganic acid salt. An organic acid is at least one species selected from the group consisting of malonic acid,
20 succinic acid, adipic acid, lactic acid, malic acid, citric acid, glycine, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, iminodiacetic acid, and fumaric acid. Meanwhile, an inorganic acid salt is at least one species selected from the group consisting
25 of sodium sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, ammonium sulfate, nickel nitrate, aluminum nitrate, ammonium nitrate, ferric nitrate, aluminum chloride, and nickel sulfamate. The amount of an organic acid or an inorganic acid salt which is incorporated into the polishing composition is preferably
30 0.003-10 wt.% on the basis of the entirety of the composition.

A polishing accelerator which may be employed in the present invention may be a combination of an organic acid and at least one of an organic acid salt and an inorganic acid salt. An organic acid is at least one species selected from the group consisting of malonic acid,
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It is employed for polishing a hard disk substrate. Polishing composition is the amount when the composition is afroremonted amount of each component in the 1.0 wt.% on the basis of the entity of the composition. Polishing rate decreases. The amount is preferably 0.01-1.0 wt.% on the basis of the amount is very large, not obtained, whereas when the amount is very small, the effect of reducing dub-off is the basis of the entity of the composition. When the amount is very small, the effect of reducing dub-off is not obtained significantly or in combination of the present employed in the polishing composition of the present invention.

The amount of HPC, HPMC, or EHCC, which is excellent polishing properties of the composition.

Preferably employed in combination so as to obtain organic acid and a salt of the same organic acid are acid salt is employed as a polishing accelerator, an acid salt is a combination of an organic acid and an organic

When a combination of an organic acid and an organic 0.003 wt.% on the basis of the entity of the composition.

the amount of an organic acid is preferably at least 1.0 wt.% on the basis of the entity of the composition. In this case, polishing composition is preferably 0.01-1.0 wt.% on the basis of the composition which is incorporated into the combination of an organic acid salt or a combination of an organic acid and an inorganic acid salt is employed, the amount of an organic acid and an inorganic acid salt is incorporated into the combination of an organic acid salt or a combination of an organic acid and an inorganic salt which is nitrate, ferric nitrate, aluminum chloride, and ammonium sulfate, nickel nitrate, aluminum sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, selected from the group consisting of sodium sulfate, combination with the organic acid is at least one species acid. An inorganic acid salt which is employed in salt, sodium salt, or ammonium salt of the above organic salt in combination with the organic acid may be a potassium and fumaric acid. An organic acid salt which is employed in combination with the organic acid, may be a potassium gluconic acid, heptogluconic acid, inosidacetic acid, citric acid, glycine, aspartic acid, tartaric acid,

succinic acid, adipic acid, lactic acid, maleic acid, 10 15 20 25 30 35

Therefore, it is efficient that the polishing composition containing each component in an amount larger than that described above is produced and transported, and the composition is diluted upon use such that the amount of 5 the component becomes as described above.

If necessary, in the polishing composition of the present invention, there may be employed, as an additive, alumina sol, a surfactant, a cleaning agent, a rust preventive, a preservative, a pH regulating agent, and a 10 surface modification agent such as sulfamic acid or phosphoric acid which is known to exhibit the effect for reducing surface defects.

The polishing composition of the present invention preferably has a pH of 2-6.

15 Examples

The present invention will next be described in more detail by way of examples, which should not be construed as limiting the invention thereto.

20 Examples 1 through 15 are shown in Table 1, and Comparative Examples 1 through 6 are shown in Table 2.

(Preparation of polishing composition)

Aluminum hydroxide was heated at about 1,200°C in air in a firing furnace, to thereby obtain α -alumina. The thus-obtained α -alumina was crushed and subjected to 25 wet-classification, thereby preparing alumina samples having mean particle sizes of 0.6 μm , 0.7 μm , and 1.0 μm .

Subsequently, on the basis of compositions shown in Tables 1 and 2, water, alumina, a polishing accelerator, and HPC, HPMC, HEMC, or EHEC were weighed, incorporated, 30 and mixed, to thereby prepare a polishing composition sample.

(Polishing conditions)

An NiP-plated aluminum disk (size: 3.5 inch) was employed as a workpiece to be polished. A polishing test 35 and evaluation of the disk were carried out under the following conditions.

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Polishing test conditions:

9B double-sided polishing machine

(product of System Setiko K.K.)

Polishing pad: Politec DG

Number of revolutions of surface plate:

Upper surface plate 28 rpm,
Lower surface plate 45 rpm,
Feed rate of slurry: 100 ml/min.

Sur gear 8 rpm

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Feed rate of slurry: 100 ml/min.

Polishing time: 5 minutes

Operation pressure: 80 g/cm²

Evaluation of disk

Polishing rate: calculated by difference in weight before and after polishing

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Quality of polished surface:

the disk

were observed under a microscope, and pits, protrusions, and scratches on disks

rating "good" was assigned when the total number of pits is not more than 10 for both sides of five disks, the total number of protrusions is 0 for both sides of five disks, and the total number of pits is not more than 5 for both sides of one disk

20

is not more than 5 for both sides of one disk, and the total number of scratches

measured by use of a surfaceorder (model: SE-30D, product of Kosaka Keikgyo)

Amount of dub-off:

disk

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is not more than 5 for both sides of one disk

measured as shown in fig. 1)

The results of polishing test of Examples and Comparative Examples are shown in Tables 1 and 2,

30

respectively.

Table 1

Ex.	α -Alumina		Polishing accelerator				Evaluation of polishing		
	Particle size D_{50}	Amount	Organic acid		Organic acid salt/inorganic acid salt		HPC/HRRC	Polishing rate	Surface defect
			μ_m	%	Type	%			
1	0.7	6	Lactic acid	0.5	Sodium lactate	1.0	HPC 0.1	1.13	Good
2	0.6	6	Lactic acid	0.5	Sodium lactate	1.0	HPC 0.1	0.78	Good
3	0.7	6	Lactic acid	4.0	Sodium lactate	5.0	HPC 1.0	1.15	Good
4	1.0	6	Malic acid	0.7	Sodium malate	0.2	HPC 0.1	1.35	Good
5	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPC 0.1	1.24	Good
6	0.6	6	Malic acid	0.7	Sodium malate	0.2	HPC 0.1	0.88	Good
7	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPC 1.0	1.04	Good
8	0.7	6	Malic acid	5.0	Sodium malate	4.0	HPC 1.0	1.25	Good
9	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPMC 0.1	1.22	Good
10	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPMC 0.1	1.21	Good
11	0.7	6	Gluconic acid	0.5	Sodium gluconate	0.5	HPC 0.1	0.98	Good
12	0.6	6	Gluconic acid	0.5	Sodium gluconate	0.5	HPC 0.1	0.73	Good
13	0.7	6	Lactic acid	0.5	-	-	HPC 0.1	1.02	Good
14	0.7	6	Malic acid	0.7	Nickel sulfate	0.3	HPC 0.1	1.09	Good
15	0.7	6	-	-	Aluminum nitrate	1.0	HPC 0.1	1.13	Good

Table 2

Comp. Ex.	α-Alumina		Polishing accelerator		Evaluation of polishing			
	Particle size D ₅₀	Amount	Organic acid	Organic acid salt/inorganic acid salt	HPC/HRRC	Polishing rate	Surface defect	Amount of dub-off
1	0.7	6	Lactic acid	0.5	Sodium lactate	1.0	0	1.18
2	1.0	6	Malic acid	0.7	Sodium malate	0.2	0	1.27
3	0.7	6	Malic acid	0.7	Sodium malate	0.2	0	1.18
4	0.6	6	Malic acid	0.7	Sodium malate	0.2	0	0.93
5	0.7	6	Gluconic acid	0.5	Sodium gluconate	0.5	0	1.08
6	0.6	6	Gluconic acid	0.5	Sodium gluconate	0.5	0	0.81

As is apparent from comparison of Table 1 with Table 2, when HPC, HPMC, or HEMC is incorporated into a polishing composition, the amount of dub-off is reduced; i.e., the composition is improved.

5 Industrial Applicability

As described above, the polishing composition of the present invention comprising water, alumina, a polishing accelerator, and at least one of HPC and HPMC enables maintenance of a predetermined polishing rate, surface 10 accuracy, and mirror surface without surface defects, and can provide excellent polishing performance so as to reduce the amount of dub-off.

1. A poliashating composition comprising water, a poliashating material, a poliashating accelerator, and at least one of hydroxypyropyl cellulose and hydroxylalkyl alkyl cellulose.

2. A poliashating composition comprising to claim 1, wherein the poliashating material is selected from among alumina, sillica, titania, zirconia, and ceria.

3. A poliashating composition according to claim 1, wherein the poliashating material is alumina.

4. A poliashating composition according to any one of claims 1, 2 and 3, wherein the poliashating accelerator comprises an organic acid or an inorganic acid salt.

5. A poliashating composition according to any one of claims 1, 2, and 3, wherein the poliashating accelerator comprises an organic acid and at least one of organic acids, succinic acid, adipic acid, lactic acid, maleic acid, citric acid, glycinic acid, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, itaconidacetic acid, and fumaric acid.

6. A poliashating composition according to any one of claims 1 through 5, wherein the organic acid is at least one species selected from the group consisting of acetic acid, succinic acid, adipic acid, lactic acid, maleic acid, citric acid, glycinic acid, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, itaconidacetic acid, and fumaric acid.

7. A poliashating composition according to any one of claims 4 through 6, wherein the inorganic acid salt is at least one species selected from the group consisting of sodium sulfite, magnesium sulfite, nickel sulfite, aluminum sulfite, ammonium sulfite, ferric nitrate, aluminum nitrate, ammonium nitrate, ammonium sulfite, sodium sulfite, and nickel sulfite.

8. A poliashating composition according to claim 5, wherein the organic acid salt is a potassium salt, a sodium salt, or an ammonium salt of the organic acid.

9. A poliashating composition according to any one of claims 1 through 8, wherein the amount of the poliashating

CLAIMS

accelerator is 0.01-10 wt.% on the basis of the entirety of the composition.

10. A polishing composition according to any one of claims 1 through 9, wherein the hydroxyalkyl alkyl cellulose is at least one species selected from the group consisting of hydroxypropyl methyl cellulose, hydroxyethyl methyl cellulose, and ethyl hydroxyethyl cellulose.

10 11. A polishing composition according to any one of claims 1 through 10, wherein the amount of hydroxypropyl cellulose and/or hydroxyalkyl alkyl cellulose is 0.001-2 wt.% on the basis of the entirety of the composition.

15 12. A method for a precision polishing, comprising polishing a workpiece with a polishing composition comprising water, a polishing material, a polishing accelerator, and at least one of hydroxypropyl cellulose and hydroxyalkyl alkyl cellulose.

20 13. A method according to claim 12, wherein said workpiece is an aluminum magnetic disk substrate.

20 14. A polishing composition according to claim 12 or 13, wherein the polishing material is selected from among alumina, silica, titania, zirconia, and ceria.

25 15. A polishing composition according to claim 12 or 13, wherein the polishing material is alumina.

16. A polishing composition according to any one of claims 12 through 14, wherein the polishing accelerator comprises an organic acid or an inorganic acid salt.

30 17. A polishing composition according to any one of claims 12 through 16, wherein the polishing accelerator comprises an organic acid and at least one of an organic acid salt and an inorganic acid salt.

35 18. A polishing composition according to any one of claims 12 through 17, wherein the organic acid is at least one species selected from the group consisting of malonic acid, succinic acid, adipic acid, lactic acid, malic acid, citric acid, glycine, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, iminodiacetic

19. A poliisahing composition according to any one of claims 16 through 18, wherein the inorganic acid salt is at least one species selected from the group consisting of sodium sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, ammonium sulfate, ferric nitrate, aluminum nitrate, ammonium nitrate, ferric nitrate, and sodium chloride, and nickel sulfamate.

20. A poliisahing composition according to claim 17, wherein the organic acid salt is a potassium salt, a sodium salt, or an ammonium salt of the organic acid as recited in claim 18.

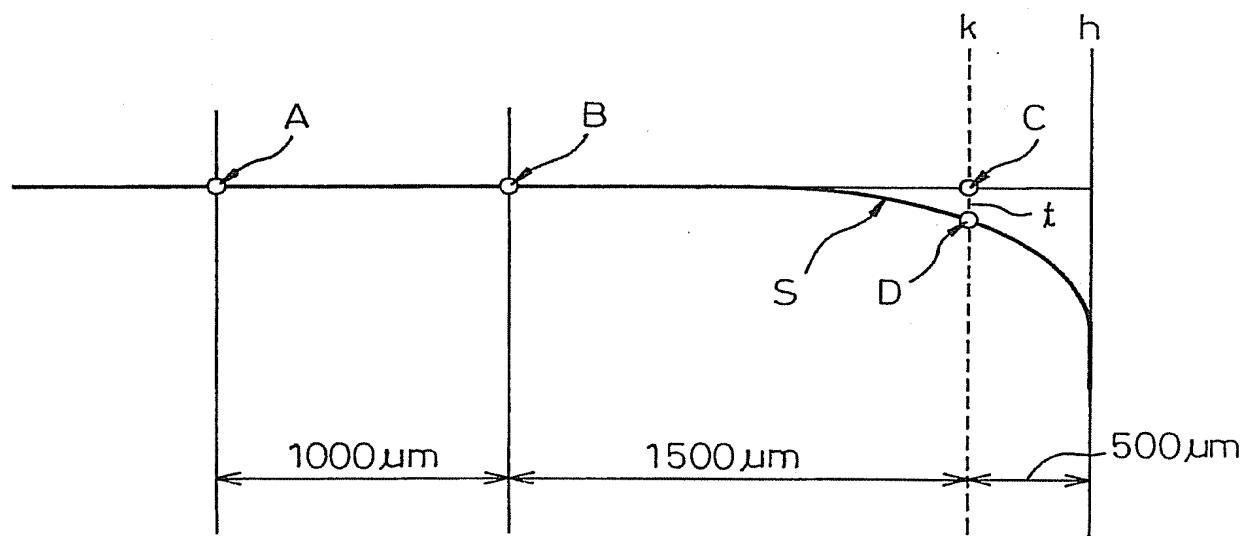
21. A poliisahing composition according to any one of claims 12 through 20, wherein the amount of the poliisahing accelerator is 0.01-10 wt.-% on the basis of the poliisahing composition.

22. A poliisahing composition according to any one of claims 12 through 21, wherein the hydroxyethyl alkyl cellulose is at least one species selected from the group consisting of hydroxypropyl methyl cellulose, and ethyl hydroxyethyl cellulose.

23. A poliisahing composition according to any one of claims 12 through 22, wherein the amount of hydroxypropyl cellulose and/or hydroxyethyl alkyl cellulose is 0.001-2 wt.-% on the basis of the entirety of the composition.

1/1

Fig.1



A. CLASSIFICATION OF SUBJECT MATTER		IPC 7 C09G C09K C23F	
According to International Patent Classification (IPC) or to both national classification and IPC		Minimum documental search (classification system followed by classification symbols)	
B. FIELDS SEARCHED		Documental search other than minimum documentation to the extent that such documents are included in the fields searched	
Electronic database consulted during the international search (name of data base and, where practical, search terms used)		MPI Data, PAJ, EPO-International	
C. DOCUMENTS CONSIDERED TO BE RELEVANT		Category of document, with indication, where applicable, of the relevant passages	
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P, X		Y	
Category		Citation of document, with indication, where applicable, of the relevant passages	
C. DOCUMENTS CONSIDERED TO BE RELEVANT		Relevant to claim No.	
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Y		Further documents are listed in the continuation of box C.	
X		Special categories of cited documents:	
A. documents relied on after the international filing date or priority date and not in conflict with the theory underlying the invention which may throw doubts on priority claim(s) or cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone X. document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone Y. document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone Z. later document published after the international filing date or priority date and not in conflict with the theory underlying the invention which may throw doubts on priority claim(s) or cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone		E. earlier document relied on before the international filing date or priority date and not in conflict with the theory underlying the invention which may throw doubts on priority claim(s) or cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone X. document published on or after the international filing date which is cited to establish the publication date of another document or other reason (as specified)	
B. documents relied on before the international filing date but in the art which is cited to establish the publication date of another document or other reason (as specified)		Z. documents published prior to the international filing date but in the art which is cited to establish the publication date of another document or other reason (as specified)	
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E. documents relied on before the international filing date but in the art which is cited to establish the publication date of another document or other reason (as specified)		Date of mailing of the international search report	
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F. Date of the actual completion of the international search		Name and mailing address of the ISA	
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INTERNATIONAL SEARCH REPORT

International Application No
PCT/JP 00/06805

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Int'l. Journal Application No	Information on patent family members		
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Patent document	Publication date	Patent family member(s)	Publication date

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